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Shaped Bodies of Detergent with Improved Properties

This invention relates to highly compacted detergents which are produced in predetermined three-dimensional forms. More particularly, the invention relates to shaped bodies of detergents which are used for washing textiles in a domestic washing machine and which are referred to in short as detergent tablets.

Nowadays, detergents are commercially available in the form of liquid or solid products. Solid products can be formulated as conventional powders or as concentrates obtainable, for example, by granulation or extrusion. Concentrated detergents have the advantage over conventional powders that they are easier and less expensive to package and can be used in smaller doses per wash cycle. The reduced pack sizes also reduce transportation and storage costs. The most highly concentrated form in which detergents are now commercially available in some countries are tablets. Whereas water softeners and dishwashing detergents are widely available in tablet form, tabletted laundry detergents pose various problems which, hitherto, have prevented their use on a wide scale and have been an obstacle to consumer acceptance. In view of the distinctly higher surfactant contents, the problems normally occurring with tablets are aggravated. Detergent tablets containing alkoxylated nonionic surfactants are particularly problematical because surfactants belonging to this class have an adverse effect on the solubility of the tablets. On the other hand, the surfactants in question are particularly desirable by virtue of their high cleaning performance.

In particular, the dichotomy between a sufficiently hard tablet and a sufficiently fast disintegration time is a central problem. Since sufficiently stable, i.e. dimensionally stable and fracture-resistant, tablets can only be produced by applying relatively high tableting pressures, the tablet

ingredients are heavily compacted which delays disintegration of the tablet in the aqueous wash liquor and hence leads to excessively slow release of the active substances in the washing process. The delayed disintegration of the tablets has the further disadvantage that conventional detergent  
5 tablets cannot be flushed into the washing process from the dispensing compartment of domestic washing machines because the tablets do not disintegrate sufficiently quickly into secondary particles which are small enough to be flushed into the drum of the washing machine from the dispensing compartment.

10 Many proposals have been put forward in the prior art with a view to overcoming the dichotomy between hardness, i.e. transportation and handling stability, and easy disintegration of the tablets. One proposed solution which is known in particular from the pharmaceutical field and which has been extended to detergent tablets is to incorporate certain  
15 disintegration aids which facilitate the access of water or which have a swelling or effervescing or other disintegrating effect on contact with water. Other proposed solutions from the patent literature include the tableting of premixes with certain particle sizes, the separation of individual ingredients from certain other ingredients and the coating of individual ingredients or  
20 the tablet as a whole with binders.

Thus, **EP-A-0 522 766** (Unilever) describes tablets of a compacted particulate detergent composition containing surfactants, builders and disintegration aids (for example based on cellulose), the particles being at least partly coated with the disintegration aid which shows both a binder  
25 effect and a disintegrating effect during dissolution of the tablets in water. This document also refers to the general difficulty of producing tablets combining adequate stability with good solubility. The particle size of the mixture to be tableted is said to be above 200  $\mu\text{m}$ , the upper and lower limits to the individual particle sizes differing by no more than 700  $\mu\text{m}$  from  
30 one another.

Other documents concerned with the production of detergent tablets are **EP-A-0 716 144** (Unilever), which describes tablets with an outer coating of water-soluble material, and **EP-A-0 711 827** (Unilever) which mentions a citrate of defined solubility as an ingredient.

5           The use of binders which optionally develop a disintegrating effect (more particularly polyethylene glycol) is disclosed in **EP-A-0 711 828** (Unilever) which describes detergent tablets obtained by tableting a particulate detergent composition at temperatures of 28°C to the melting point of the binder, the tableting process always being carried out below  
10       the melting temperature. It is clear from the Examples of this document that the tablets produced in accordance with its teaching have higher fracture resistances when tableting is carried out at elevated temperature.

Detergent tablets in which individual ingredients are separated from others are described, for example, in **EP-A-0 481 793** (Unilever). The  
15       detergent tablets disclosed in this document contain sodium percarbonate which is separated from all other components that could affect its stability.

Earlier German patent application **197 54 289.1** (Henkel) describes detergent tablets combining high hardness with a favorable disintegration and solubility profile which are obtainable by incorporation of alkyl  
20       polyglycosides (APGs) in the tablets. According to the teaching of this document, APG contents above 0.2% by weight, based on the tablet, are preferred.

None of the cited prior art documents concerned with detergent tablets suggests surfactant combinations which improve the property profile  
25       of the tablets in regard to hardness and disintegration time. None of the documents cited above is concerned with improving the solubility of detergent tablets containing nonionic surfactants by the selective use of other classes of surfactants to compensate for the negative effects of the alkoxyated nonionic surfactants.

30           Accordingly, the problem addressed by the present invention was to

provide detergent tablets which would contain alkoxylated nonionic surfactants, but which would still combine high hardness with excellent disintegration properties.

5 It has now been found that the negative effects of alkoxylated nonionic surfactants on the hardness and disintegration time of detergent tablets can be overcome if nonionic surfactants from the class of sugar surfactants are incorporated in the tablets in a certain ratio to the alkoxylated nonionic surfactants.

10 The present invention relates to detergent tablets of compacted particulate detergent containing surfactant(s), builder(s) and optionally other detergent ingredients, characterized in that the tablets contain

a) 0.2 to 10% by weight of one or more nonionic surfactants from the group of alkyl polyglycosides and/or polyhydroxyfatty acid amides and

15 b) 1 to 15% by weight of one or more nonionic surfactants from the group of alkoxylated alcohols

in a ratio of 10:1 to 1:10.

According to the invention, detergent tablets in which the total content of nonionic surfactants, i.e. surfactants from groups a) and b), is between 1 and 15% by weight are preferred.

20 The ratio of sugar surfactants a) to alkoxylates b) may be varied as required within the described limits. Whereas, in principle, any ratio of a) to b) between 10:1 and 1:10 is possible and works in accordance with the invention, detergent tablets in which the ratio of nonionic surfactants from the group of alkyl polyglycosides and/or glucamides to nonionic surfactants  
25 from the group of alkoxylated alcohols is 2:1 to 1:8, preferably 1:1 to 1:7 and more preferably 1:2 to 1:4 are preferred for reasons of cost.

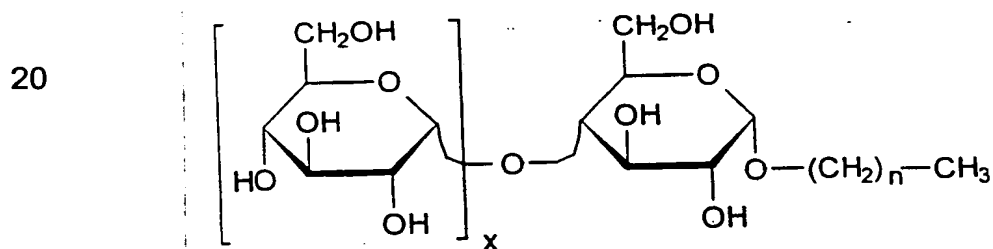
The nonionic surfactants of category a) emanate from the group of alkyl polyglycosides and/or glucamides. They are used in quantities of 0.2 to 10% by weight in the detergent tablets according to the invention,  
30 detergent tablets containing 0.2 to 8% by weight, preferably 0.5 to 5% by

weight and more preferably 1 to 3% by weight of one or more nonionic surfactants from the group of alkyl polyglycosides and/or glucamides being preferred.

Alkyl polyglycosides suitable for use in accordance with the invention correspond to the general formula  $RO(G)_z$  where R is a linear or branched, more particularly 2-methyl-branched, saturated or unsaturated aliphatic radical containing 8 to 22 and preferably 12 to 18 carbon atoms and G stands for a glucose unit containing 5 or 6 carbon atoms, preferably glucose. The degree of glycosidation z is between 1.0 and 4.0, preferably between 1.0 and 2.0 and more preferably between 1.1 and 1.4.

According to the invention, detergent tablets containing alkyl polyglycoside(s) with a degree of glycosidation of 1.0 to 4.0, preferably 1.0 to 2.0 and more preferably 1.1 to 1.4 as nonionic surfactant(s) are preferred.

Linear alkyl polyglucosides, i.e. alkyl polyglycosides in which the polyglycosyl unit is a glucose unit and the alkyl group is an n-alkyl group, are preferably used. They may be described by the following formula:



25 in which x is the degree of glycosidation z described above reduced by 1, so that preferred values for x are between 0 and 3, preferably between 0 and 1 and more preferably between 0.1 and 0.4. The number n of methylene groups is preferably between 7 and 21 and more preferably between 11 and 17 carbon atoms.

30 Preferred detergent tablets contain alkyl polyglucoside(s), i.e. APGs,

in which the polyglycosyl unit is a glucose unit, as nonionic surfactant(s) a).

The alkyl polyglycosides used in accordance with the invention (hereinafter referred to as APGs) may be produced from known raw materials by known methods. For example, dextrose is reacted with n-  
5 butanol in the presence of an acidic catalyst to form butyl polyglycoside mixtures which are trans-glycosidated with long-chain alcohols, again in the presence of an acidic catalyst, to form the required alkyl polyglycoside mixtures. Dextrose may also be directly glycosidated with the required long-chain alcohol to form the required alkyl polyglycoside mixtures.

10 The structure of the products is variable within certain limits. The alkyl group is determined by the choice of the long-chain alcohol. For economic reasons, the industrially obtainable C<sub>8-22</sub> alcohols, more particularly native alcohols from the hydrogenation of carboxylic acids or carboxylic acid derivatives, are preferred. The alcohols obtainable from  
15 industrial-scale alcohol syntheses, such as oxo alcohols and Ziegler alcohols, may also be used.

The polyglycosyl units G<sub>y</sub> are determined on the one hand by the choice of the carbohydrate and on the other hand by the adjustment of the average degree of polymerization (degree of glycosidation y), as described  
20 for example in DE 19 43 689. It is known that, in principle, polysaccharides, for example starches, maltodextrins, dextrose, galactose, mannose, xylose, etc., may be used. The industrially obtainable carbohydrates starch, maltodextrins and, above all, dextrose are preferred. Since the economically interesting APG syntheses are not regio- and  
25 stereoselective, the alkyl polyglycosides are always mixtures of oligomers which, in turn, represent mixtures of various isomeric forms. They are present alongside one another with α- and β-glycosidic bonds in pyranose and furanose form. The linkage points between two saccharide units are also different.

30 Alkyl polyglycosides suitable for use in accordance with the

invention may also be produced by mixing alkyl polyglycosides with alkyl monoglycosides. Alkyl monoglycosides may be obtained from alkyl polyglycosides and concentrated, for example by the process disclosed in **EP 092 355** using polar solvents, such as acetone. The degree of glycosidation of alkyl polyglycosides is normally determined by  $^1\text{H}$ -nuclear resonance measurements.

Besides alkyl polyglycosides, glucamides may also be used as category a) nonionic surfactants. In the context of the present invention, "glucamides" are any polyhydroxyfatty acid amides which can be described by formula (I):



in which RCO is an aliphatic acyl group containing 6 to 22 carbon atoms,  $\text{R}^1$  is hydrogen, an alkyl or hydroxyalkyl group containing 1 to 4 carbon atoms and [Z] is a linear or branched polyhydroxyalkyl group containing 3 to 10 carbon atoms and 3 to 10 hydroxyl groups. The polyhydroxyfatty acid amides are known substances which may normally be obtained by reductive amination of a reducing sugar with ammonia, an alkylamine or an alkanolamine and subsequent acylation with a fatty acid, a fatty acid alkyl ester or a fatty acid chloride.

Preferred detergent tablets contain polyhydroxyfatty acid amides corresponding to formula (I):



in which RCO is an aliphatic acyl group containing 6 to 22 carbon atoms,  $\text{R}^1$  is hydrogen, an alkyl or hydroxyalkyl group containing 1 to 4 carbon atoms and [Z] is a linear or branched polyhydroxyalkyl group containing 3

to 10 carbon atoms and 3 to 10 hydroxyl groups, as nonionic surfactant(s)  
a).

Polyhydroxyfatty acid amides corresponding to formula (I), in which  
the acyl group RCO has a relatively narrow chain distribution and [Z] is  
5 selected from typical sugar units are preferably used. Accordingly,  
preferred detergent tablets according to the invention are characterized in  
that they contain a polyhydroxyfatty acid amide corresponding to formula (I)  
in which RCO is an aliphatic acyl group containing 12 to 18 carbon atoms,  
R<sup>1</sup> is hydrogen, an alkyl or hydroxyalkyl group containing 1 to 4 carbon  
10 atoms and [Z] stands for a glucose, fructose, maltose, lactose, galactose,  
mannose or xylose unit.

In one particularly preferred embodiment of the invention, the  
generic term "glucamides" does not stand for polyhydroxyfatty acid amides  
in general, but for the class of glucamides, i.e. compounds corresponding  
15 to formula I in which [Z] derives from glucose. Particularly preferred  
detergent tablets are characterized by the presence of a C<sub>12-18</sub>-N-methyl  
glucamide (R<sup>1</sup> = CH<sub>3</sub>, Z = glucose unit in formula I).

The group of polyhydroxyfatty acid amides, which are referred to  
simply as glucamides in the context of the present invention, also includes  
20 compounds corresponding to formula (II):



25 in which R is a linear or branched alkyl or alkenyl group containing 7 to 12  
carbon atoms, R<sup>1</sup> is a linear, branched or cyclic alkyl group or an aryl group  
containing 2 to 8 carbon atoms and R<sup>2</sup> is a linear, branched or cyclic alkyl  
group or an aryl group or an oxyalkyl group containing 1 to 8 carbon atoms,  
30 methyl, ethyl, propyl, isopropyl, n-butyl, isobutyl, tert. butyl or phenyl groups  
being preferred, and [Z] is a linear polyhydroxyalkyl group, of which the  
alkyl chain is substituted by at least two hydroxyl groups, or alkoxyated,



preferably ethoxylated or propoxylated, derivatives of that group.

[Z] is preferably obtained by reductive amination of a reduced sugar, for example glucose, fructose, maltose, lactose, galactose, mannose or xylose. The N-alkoxy- or N-aryloxy-substituted compounds may then be converted into the required polyhydroxyfatty acid amides by reaction with fatty acid methyl esters in the presence of an alkoxide as catalyst, for example in accordance with the teaching of International patent application **WO-A-95/07331**.

As a second group of nonionic surfactants, the detergent tablets according to the invention contain nonionic surfactant(s) from the group of alkoxylated alcohols in quantities of 1 to 15% by weight, based on the tablet.

The quantities in which category b) nonionic surfactant(s) are used are preferably within relatively narrow limits so that detergent tablets containing 1 to 12% by weight, preferably 2.5 to 10% by weight and more preferably 4 to 8% by weight of one or more nonionic surfactants from the group of alkoxylated alcohols are preferred.

Any relatively long-chain alcohols of natural or synthetic origin may be used as the alcohols which give the category b) surfactants by alkoxylation. The industrially obtainable alcohols containing 8 to 22 carbon atoms, more particularly native alcohols from the hydrogenation of carboxylic acids or carboxylic acid derivatives (so-called fatty alcohols), are preferred on economic grounds. The alcohols obtainable from industrial-scale alcohol syntheses, such as oxo alcohols and Ziegler alcohols, may also be used.

The alcohols are in particular primary alcohols preferably containing 8 to 18 carbon atoms in which the alcohol component may be linear or, preferably, methyl-branched in the 2-position or may contain linear and methyl-branched residues in the form of the mixtures typically present in oxoalcohol residues. In preferred alkoxylated nonionic surfactants, the

alcohol component is even narrower in its chain distribution so that preferred detergent tablets contain alkoxyated alcohol(s) derived from C<sub>8-22</sub> alcohols, preferably from C<sub>8-20</sub> alcohols and more preferably from C<sub>12-18</sub> alcohols as nonionic surfactant(s) b).

- 5           However, alcohol ethoxylates containing linear residues of alcohols of native origin with 12 to 18 carbon atoms, for example coconut, palm, tallow or oleyl alcohol, and on average 2 to 8 EO per mole of alcohol are particularly preferred. Preferred ethoxylated alcohols include, for example, C<sub>12-14</sub> alcohols containing 3 EO or 4 EO, C<sub>9-11</sub> alcohol containing 7 EO, 10 C<sub>13-15</sub> alcohols containing 3 EO, 5 EO, 7 EO or 8 EO, C<sub>12-18</sub> alcohols containing 3 EO, 5 EO or 7 EO and mixtures thereof, such as mixtures of C<sub>12-14</sub> alcohol containing 3 EO and C<sub>12-18</sub> alcohol containing 5 EO. The degrees of ethoxylation mentioned represent statistical mean values which, for a special product, can be a whole number or a broken number.
- 15 Preferred alcohol ethoxylates have a narrow homolog distribution (narrow range ethoxylates, NRE). In addition to these nonionic surfactants, fatty alcohols containing more than 12 EO may also be used, examples including tallow alcohol containing 14 EO, 25 EO, 30 EO or 40 EO.

- Preferred detergent tablets contain ethoxylated alcohols with 20 degrees of ethoxylation of 2.0 to 10, preferably 5.0 to 8.0 and more preferably 5.5 to 7.0 as nonionic surfactant(s) b).

- It is of course possible to use alkoxyated alcohols which not only were reacted with ethylene or propylene oxide or other alkoxyating agents, but are obtainable by simultaneous or successive reaction with several 25 alkoxyating agents (for example ethoxylation/propoxylation). Such mixed-alkoxyated nonionic surfactants may have a statistical sequence of the individual EO-PO units or may be present as block or graft polymers.

Preferred detergent tablets contain mixed-alkoxyated, preferably ethoxylated and propoxylated, alcohols as nonionic surfactant(s) b).

- 30           To develop their cleaning performance, the surfactant granules

contain surfactants from the group of anionic, nonionic, zwitterionic or cationic surfactants, anionic surfactants being distinctly preferred on economic grounds and for their performance spectrum.

Suitable anionic surfactants are, for example, those of the sulfonate and sulfate type. Suitable surfactants of the sulfonate type are preferably C<sub>9-13</sub> alkyl benzenesulfonates, olefin sulfonates, i.e. mixtures of alkene and hydroxyalkane sulfonates, and the disulfonates obtained, for example, from C<sub>12-18</sub> monoolefins with an internal or terminal double bond by sulfonation with gaseous sulfur trioxide and subsequent alkaline or acidic hydrolysis of the sulfonation products. Other suitable surfactants of the sulfonate type are the alkane sulfonates obtained from C<sub>12-18</sub> alkanes, for example by sulfochlorination or sulfoxidation and subsequent hydrolysis or neutralization. The esters of  $\alpha$ -sulfofatty acids (ester sulfonates), for example the  $\alpha$ -sulfonated methyl esters of hydrogenated coconut oil, palm kernel oil or tallow fatty acids, are also suitable.

Other suitable anionic surfactants are sulfonated fatty acid glycerol esters. Fatty acid glycerol esters in the context of the present invention are the monoesters, diesters and triesters and mixtures thereof which are obtained where production is carried out by esterification of a monoglycerol with 1 to 3 moles of fatty acid or in the transesterification of triglycerides with 0.3 to 2 moles of glycerol. Preferred sulfonated fatty acid glycerol esters are the sulfonation products of saturated fatty acids containing 6 to 22 carbon atoms, for example caproic acid, caprylic acid, capric acid, myristic acid, lauric acid, palmitic acid, stearic acid or behenic acid.

Preferred alk(en)yl sulfates are the alkali metal salts and, in particular, the sodium salts of the sulfuric acid semiesters of C<sub>12-18</sub> fatty alcohols, for example cocofatty alcohol, tallow fatty alcohol, lauryl, myristyl, cetyl or stearyl alcohol, or C<sub>10-20</sub> oxoalcohols and the corresponding semiesters of secondary alcohols with the same chain length. Other preferred alk(en)yl sulfates are those with the chain length mentioned

which contain a synthetic, linear alkyl chain based on a petrochemical and which are similar in their degradation behavior to the corresponding compounds based on oleochemical raw materials. C<sub>12-16</sub> alkyl sulfates, C<sub>12-15</sub> alkyl sulfates and C<sub>14-15</sub> alkyl sulfates are preferred from the point of view of washing technology. Other suitable anionic surfactants are 2,3-alkyl sulfates which may be produced, for example, in accordance with **US 3,234,258** or **US 5,075,041** and which are commercially obtainable as products of the Shell Oil Company under the name of DAN®.

The sulfuric acid monoesters of linear or branched C<sub>7-21</sub> alcohols ethoxylated with 1 to 6 moles of ethylene oxide, such as 2-methyl-branched C<sub>9-11</sub> alcohols containing on average 3.5 moles of ethylene oxide (EO) or C<sub>12-18</sub> fatty alcohols containing 1 to 4 EO, are also suitable. In view of their high foaming capacity, they are only used in relatively small quantities, for example in quantities of 1 to 5% by weight, in dishwashing detergents.

Other suitable anionic surfactants are the salts of alkyl sulfosuccinic acid which are also known as sulfosuccinates or as sulfosuccinic acid esters and which represent monoesters and/or diesters of sulfosuccinic acid with alcohols, preferably fatty alcohols and, more particularly, ethoxylated fatty alcohols. Preferred sulfosuccinates contain C<sub>8-18</sub> fatty alcohol residues or mixtures thereof. Particularly preferred sulfosuccinates contain a fatty alcohol residue derived from ethoxylated fatty alcohols which, considered in isolation, represent nonionic surfactants (for a description, see below). Of these sulfosuccinates, those of which the fatty alcohol residues are derived from narrow-range ethoxylated fatty alcohols are particularly preferred. Alk(en)yl succinic acid preferably containing 8 to 18 carbon atoms in the alk(en)yl chain or salts thereof may also be used.

Other suitable anionic surfactants are, in particular, soaps. Suitable soaps are saturated fatty acid soaps, such as the salts of lauric acid, myristic acid, palmitic acid, stearic acid, hydrogenated erucic acid and behenic acid, and soap mixtures derived in particular from natural fatty

acids, for example coconut oil, palm kernel oil or tallow fatty acids.

The anionic surfactants, including the soaps, may be present in the form of their sodium, potassium or ammonium salts and as soluble salts of organic bases, such as mono-, di- or triethanolamine. The anionic  
5 surfactants are preferably present in the form of their sodium or potassium salts and, more preferably, in the form of their sodium salts.

So far as the choice of anionic surfactants used in the detergent tablets according to the invention is concerned, there are no basic requirements to restrict the freedom of formulation. However, preferred  
10 surfactant tablets do have a soap content in excess of 0.2% by weight, based on the total weight of the detergent tablet. Preferred anionic surfactants are alkyl benzenesulfonates and fatty alcohol sulfates.

Besides the deterative substances, builders are the most important ingredients of detergents. The detergent tablets according to the invention  
15 may contain any of the builders typically used in detergents and cleaners, i.e. in particular zeolites, silicates, carbonates, organic cobuilders and - providing there are no ecological objections to their use - also the phosphates.

Suitable crystalline layered sodium silicates correspond to the  
20 general formula  $\text{NaMSi}_x\text{O}_{2x+1} \cdot y \text{H}_2\text{O}$ , where M is sodium or hydrogen, x is a number of 1.9 to 4 and y is a number of 0 to 20, preferred values for x being 2, 3 or 4. Crystalline layered silicates such as these are described, for example, in European patent application **EP-A-0 164 514**. Preferred crystalline layered silicates corresponding to the above formula are those in  
25 which M is sodium and x assumes the value 2 or 3. Both  $\beta$ - and  $\delta$ -sodium disilicates  $\text{Na}_2\text{Si}_2\text{O}_5 \cdot y \text{H}_2\text{O}$  are particularly preferred,  $\beta$ -sodium disilicate being obtainable, for example, by the process described in International patent application **WO-A- 91/08171**.

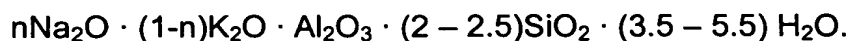
Other useful builders are amorphous sodium silicates with a  
30 modulus ( $\text{Na}_2\text{O}:\text{SiO}_2$  ratio) of 1:2 to 1:3.3, preferably 1:2 to 1:2.8 and more

preferably 1:2 to 1:2.6 which dissolve with delay and exhibit multiple wash cycle properties. The delay in dissolution in relation to conventional amorphous sodium silicates can have been obtained in various ways, for example by surface treatment, compounding, compacting or by overdrying.

5 In the context of the invention, the term "amorphous" is also understood to encompass "X-ray amorphous". In other words, the silicates do not produce any of the sharp X-ray reflexes typical of crystalline substances in X-ray diffraction experiments, but at best one or more maxima of the scattered X-radiation which have a width of several degrees of the  
10 diffraction angle. However, particularly good builder properties may even be achieved where the silicate particles produce crooked or even sharp diffraction maxima in electron diffraction experiments. This may be interpreted to mean that the products have microcrystalline regions between 10 and a few hundred nm in size, values of up to at most 50 nm  
15 and, more particularly, up to at most 20 nm being preferred. So-called X-ray amorphous silicates such as these, which also dissolve with delay in relation to conventional waterglasses, are described for example in German patent application **DE-A-44 00 024**. Compacted amorphous silicates, compounded amorphous silicates and overdried X-ray-amorphous  
20 silicates are particularly preferred.

The finely crystalline, synthetic zeolite containing bound water used in accordance with the invention is preferably zeolite A and/or zeolite P. Zeolite MAP® (Crosfield) is a particularly preferred P-type zeolite. However, zeolite X and mixtures of A, X and/or P are also suitable.  
25 According to the invention, it is preferred to use, for example, a commercially obtainable co-crystallizate of zeolite X and zeolite A (ca. 80% by weight zeolite X) which is marketed by CONDEA Augusta S.p.A. under the name of VEGOBOND AX® and which may be described by the following formula:

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The zeolite may be used both as a builder in a granular compound and for "powdering" the entire mixture to be tabletted, both these options normally being used to incorporate the zeolite in the premix. Suitable zeolites have a mean particle size of less than 10  $\mu\text{m}$  (volume distribution, as measured by the Coulter Counter Method) and contain preferably 18 to 22% by weight and more preferably 20 to 22% by weight of bound water.

The generally known phosphates may of course also be used as builders providing their use should not be avoided on ecological grounds. The sodium salts of the orthophosphates, the pyrophosphates and especially the tripolyphosphates are particularly suitable.

Useful organic builders are, for example, the polycarboxylic acids usable, for example, in the form of their sodium salts, such as citric acid, adipic acid, succinic acid, glutaric acid, tartaric acid, sugar acids, amino-carboxylic acids, nitrilotriacetic acid (NTA), providing its use is not ecologically unsafe, and mixtures thereof. Preferred salts are the salts of the polycarboxylic acids, such as citric acid, adipic acid, succinic acid, glutaric acid, tartaric acid, sugar acids and mixtures thereof.

In order to facilitate the disintegration of heavily cleaning compacted tablets, disintegration aids, so-called tablet disintegrators, may be incorporated in them to shorten their disintegration times. According to Römpp (9th Edition, Vol. 6, page 4440) and Voigt "*Lehrbuch der pharmazeutischen Technologie*" (6th Edition, 1987, pages 182-184), tablet disintegrators or disintegration accelerators are auxiliaries which promote the rapid disintegration of tablets in water or gastric juices and the release of the pharmaceuticals in an absorbable form.

These substances, which are also known as "disintegrators" by virtue of their effect, are capable of undergoing an increase in volume on contact with water so that, on the one hand, their own volume is increased

(swelling) and, on the other hand, a pressure can be generated through the release of gases which causes the tablet to disintegrate into relatively small particles. Well-known disintegration aids are carbonate/citric acid systems, although other organic acids may also be used. Swelling disintegration  
5 aids are, for example, synthetic polymers, such as polyvinyl pyrrolidone (PVP), or natural polymers and modified natural substances, such as cellulose and starch and derivatives thereof, alginates or casein derivatives.

Preferred cleaning tablets contain 0.5 to 10% by weight, preferably 3 to 7% by weight and more preferably 4 to 6% by weight of one or more  
10 disintegration aids, based on tablet weight.

According to the invention, preferred disintegrators are cellulose-based disintegrators, so that preferred cleaning tablets contain a cellulose-based disintegrator in quantities of 0.5 to 10% by weight, preferably 3 to 7% by weight and more preferably 4 to 6% by weight. Pure cellulose has  
15 the formal empirical composition  $(C_6H_{10}O_5)_n$  and, formally, is a  $\beta$ -1,4-polyacetal of cellobiose which, in turn, is made up of two molecules of glucose. Suitable celluloses consist of ca. 500 to 5000 glucose units and, accordingly, have average molecular weights of 50,000 to 500,000. According to the invention, cellulose derivatives obtainable from cellulose  
20 by polymer-analog reactions may also be used as cellulose-based disintegrators. These chemically modified celluloses include, for example, products of esterification or etherification reactions in which hydroxy hydrogen atoms have been substituted. However, celluloses in which the hydroxy groups have been replaced by functional groups that are not  
25 attached by an oxygen atom may also be used as cellulose derivatives. The group of cellulose derivatives includes, for example, alkali metal celluloses, carboxymethyl cellulose (CMC), cellulose esters and ethers and aminocelluloses. The cellulose derivatives mentioned are preferably not used on their own, but rather in the form of a mixture with cellulose as  
30 cellulose-based disintegrators. The content of cellulose derivatives in



mixtures such as these is preferably below 50% by weight and more preferably below 20% by weight, based on the cellulose-based disintegrator. In one particularly preferred embodiment, pure cellulose free from cellulose derivatives is used as the cellulose-based disintegrator.

5           The cellulose used as disintegration aid is preferably not used in fine-particle form, but is converted into a coarser form, for example by granulation or compacting, before it is added to and mixed with the premixes to be tableted. Detergent tablets which contain granular or optionally co-granulated disintegrators are described in German patent  
10 applications **DE 197 09 991** (Stefan Herzog) and **DE 197 10 254** (Henkel) and in International patent application **PCT/EP98/01203** (Henkel). Further particulars of the production of granulated, compacted or co-granulated cellulose disintegrators can also be found in these patent applications. The particle sizes of such disintegration aids are mostly above 200  $\mu\text{m}$ ,  
15 preferably at least 90% by weight of the particles being between 300 and 1600  $\mu\text{m}$  in size and, more particularly, between 400 and 1200  $\mu\text{m}$  in size. According to the invention, the above-described relatively coarse-particle cellulose-based disintegrators described in detail in the cited patent applications are preferably used as disintegration aids and are  
20 commercially obtainable, for example under the name of Arbocel® TF-30-HG from Rettenmaier.

Microcrystalline cellulose may be used as another cellulose-based disintegration aid or as part of such a component. This microcrystalline cellulose is obtained by partial hydrolysis of celluloses under conditions  
25 which only attack and completely dissolve the amorphous regions (ca. 30% of the total cellulose mass) of the celluloses, but leave the crystalline regions (ca. 70%) undamaged. Subsequent de-aggregation of the microfine celluloses formed by hydrolysis provides the microcrystalline celluloses which have primary particle sizes of ca. 5  $\mu\text{m}$  and which can be  
30 compacted, for example, to granules with a mean particle size of 200  $\mu\text{m}$ .

Cleaning tablets additionally containing a disintegration aid preferably based on cellulose, preferably in granular, cogranulated or compacted form, in quantities of 0.5 to 10% by weight, preferably in quantities of 3 to 7% by weight and more preferably in quantities of 4 to 6%  
5 by weight, based on tablet weight, represent a particularly preferred embodiment of the invention.

Cleaning tablets are produced by the application of pressure to a mixture to be tabletted which is accommodated in the cavity of a press. In the most simple method of tablet production – hereinafter referred to simply  
10 as tableting – the mixture to be tabletted is compressed directly, i.e. without preliminary granulation. The advantages of this so-called direct tableting are its simple and inexpensive application because no other process steps and hence no other items of equipment are involved. However, these advantages are offset by disadvantages. Thus, a powder  
15 mixture which is to be directly tabletted must possess adequate plastic deformability and good flow properties and must not show any tendency to separate during storage, transportation and filling of the die. Unfortunately, these three requirements are very difficult to satisfy with many mixtures so that direct tableting is often not applied, particularly in the production of  
20 detergent tablets. Accordingly, the normal method of producing detergent tablets starts out from powder-form components ("primary particles") which are agglomerated or granulated by suitable methods to secondary particles with larger particle diameters. These granules or mixtures of different granules are then mixed with individual powder-form additives and the  
25 resulting mixtures are tabletted.

According to the invention, preferred detergent tablets are obtained by tableting particulate premixes of at least one batch of surfactant-containing granules and at least one subsequently added powder-form component. It is of advantage so far as the subsequent detergent tablets  
30 are concerned if the premixes to be tabletted have a bulk density

approaching that of standard compact detergents. In one particularly preferred embodiment, the premix to be tabletted has a bulk density of at least 500 g/l, preferably of at least 600 g/l and more preferably above 700 g/l.

5 Before the particulate premix is compressed to form detergent tablets, it may be "powdered" with fine-particle surface treatment materials. This can be of advantage to the quality and physical properties of both the premix (storage, tableting) and the final detergent tablets. Fine-particle powdering materials have been known for some time in the art, zeolites,  
10 silicates and other inorganic salts generally being used. However, the premix is preferably "powdered" with fine-particle zeolite, zeolites of the faujasite type being preferred.

According to the invention, preferred detergent tablets consist of a particulate premix containing granular components and subsequently  
15 incorporated powder-form components, the, or one of the, fine-particle components subsequently incorporated being a zeolite of the faujasite type with particle sizes below 100  $\mu\text{m}$ , preferably below 10  $\mu\text{m}$  and more preferably below 5  $\mu\text{m}$  and making up at least 0.2% by weight, preferably at least 0.5% by weight and more preferably more than 1% by weight of the  
20 premix to be compressed.

Besides the constituents mentioned - surfactant, builder and disintegration aid - the detergent tablets according to the invention may contain other typical ingredients of washing/cleaning compositions from the group of bleaching agents, bleach activators, enzymes, perfumes, perfume  
25 carriers, fluorescers, dyes, foam inhibitors, silicone oils, redeposition inhibitors, optical brighteners, discoloration inhibitors, dye transfer inhibitors and corrosion inhibitors.

Among the compounds yielding  $\text{H}_2\text{O}_2$  in water which serve as bleaching agents, sodium perborate tetrahydrate and sodium perborate  
30 monohydrate are particularly important. Other useful bleaching agents are,

for example, sodium percarbonate, peroxyphosphates, citrate perhydrates and  $H_2O_2$ -yielding peracidic salts or peracids, such as perbenzoates, peroxyphthalates, diperazelaic acid, phthaliminoperacid or diperdodecane dioic acid.

5           In order to obtain an improved bleaching effect where washing is carried out at temperatures of 60°C or lower, bleach activators may be incorporated as sole constituent or as an ingredient of component b). The bleach activators may be compounds which form aliphatic peroxocarboxylic acids containing preferably 1 to 10 carbon atoms and more preferably 2 to  
10   4 carbon atoms and/or optionally substituted perbenzoic acid under perhydrolysis conditions. Substances bearing O- and/or N-acyl groups with the number of carbon atoms mentioned and/or optionally substituted benzoyl groups are suitable. Preferred bleach activators are polyacylated alkylenediamines, more particularly tetraacetyl ethylenediamine (TAED),  
15   acylated triazine derivatives, more particularly 1,5-diacetyl-2,4-dioxohexahydro-1,3,5-triazine (DADHT), acylated glycolurils, more particularly tetraacetyl glycoluril (TAGU), N-acylimides, more particularly N-nonanoyl succinimide (NOSI), acylated phenol sulfonates, more particularly n-nonanoyl or isononanoyloxybenzenesulfonate (n- or iso-NOBS), carboxylic  
20   anhydrides, more particularly phthalic anhydride, acylated polyhydric alcohols, more particularly triacetin, ethylene glycol diacetate and 2,5-diacetoxy-2,5-dihydrofuran.

          In addition to or instead of the conventional bleach activators mentioned above, so-called bleach catalysts may also be incorporated in  
25   the tablets. Bleach catalysts are bleach-boosting transition metal salts or transition metal complexes such as, for example, manganese-, iron-, cobalt-, ruthenium- or molybdenum-salen complexes or carbonyl complexes. Manganese, iron, cobalt, ruthenium, molybdenum, titanium, vanadium and copper complexes with nitrogen-containing tripod ligands  
30   and cobalt-, iron-, copper- and ruthenium-ammine complexes may also be

used as bleach catalysts.

Suitable enzymes are those from the class of proteases, lipases, amylases, cellulases or mixtures thereof. Enzymes obtained from bacterial strains or fungi, such as *Bacillus subtilis*, *Bacillus licheniformis* and *Streptomyces griseus*, are particularly suitable. Proteases of the subtilisin type are preferred, proteases obtained from *Bacillus lentus* being particularly preferred. Enzyme mixtures, for example of protease and amylase or protease and lipase or protease and cellulase or of cellulase and lipase or of protease, amylase and lipase or of protease, lipase and cellulase, but especially cellulase-containing mixtures, are of particular interest. Peroxidases or oxidases have also proved to be suitable in some cases. The enzymes may be adsorbed to supports and/or encapsulated in membrane materials to protect them against premature decomposition. The percentage content of the enzymes, enzyme mixtures or enzyme granules in the tablets according to the invention may be, for example, from about 0.1 to 5% by weight and is preferably from 0.1 to about 2% by weight.

In addition, the detergent tablets according to the invention may also contain components with a positive effect on the removability of oil and fats from textiles by washing (so-called soil repellents). This effect becomes particularly clear when a textile which has already been repeatedly washed with a detergent according to the invention containing this oil- and fat-dissolving component is soiled. Preferred oil- and fat-dissolving components include, for example, nonionic cellulose ethers, such as methyl cellulose and methyl hydroxypropyl cellulose containing 15 to 30% by weight of methoxyl groups and 1 to 15% by weight of hydroxypropoxyl groups, based on the nonionic cellulose ether, and the polymers of phthalic acid and/or terephthalic acid known from the prior art or derivatives thereof, more particularly polymers of ethylene terephthalates and/or polyethylene glycol terephthalates or anionically and/or nonionically modified derivatives

thereof. Of these, the sulfonated derivatives of phthalic acid and terephthalic acid polymers are particularly preferred.

The tablets may contain derivatives of diaminostilbenedisulfonic acid or alkali metal salts thereof as optical brighteners. Suitable optical  
5 brighteners are, for example, salts of 4,4'-bis-(2-anilino-4-morpholino-1,3,5-triazinyl-6-amino)-stilbene-2,2'-disulfonic acid or compounds of similar composition which contain a diethanolamino group, a methylamino group, an anilino group or a 2-methoxyethylamino group instead of the morpholino group. Brighteners of the substituted diphenyl styryl type, for example  
10 alkali metal salts of 4,4'-bis-(2-sulfostyryl)-diphenyl, 4,4'-bis-(4-chloro-3-sulfostyryl)-diphenyl or 4-(4-chlorostyryl)-4'-(2-sulfostyryl)-diphenyl, may also be present. Mixtures of the brighteners mentioned above may also be used.

Dyes and perfumes are added to the detergent tablets according to  
15 the invention to improve the aesthetic impression created by the products and to provide the consumer not only with the required washing performance but also with a visually and sensorially "typical and unmistakable" product. Suitable perfume oils or perfumes include individual perfume compounds, for example synthetic products of the ester,  
20 ether, aldehyde, ketone, alcohol and hydrocarbon type. Perfume compounds of the ester type are, for example, benzyl acetate, phenoxyethyl isobutyrate, p-tert.butyl cyclohexyl acetate, linalyl acetate, dimethyl benzyl carbonyl acetate, phenyl ethyl acetate, linalyl benzoate, benzyl formate, ethyl methyl phenyl glycinate, allyl cyclohexyl propionate, styrallyl  
25 propionate and benzyl salicylate. The ethers include, for example, benzyl ethyl ether; the aldehydes include, for example, the linear alkanals containing 8 to 18 carbon atoms, citral, citronellal, citronellyloxyacetaldehyde, cyclamen aldehyde, hydroxycitronellal, lillial and bourgeonal; the ketones include, for example, the ionones,  $\alpha$ -isomethyl ionone and methyl  
30 cedryl ketone; the alcohols include anethol, citronellol, eugenol, geraniol,

linalool, phenyl ethyl alcohol and terpeneol and the hydrocarbons include, above all, the terpenes, such as limonene and pinene. However, mixtures of various perfumes which together produce an attractive perfume note are preferably used. Perfume oils such as these may also contain natural  
5 fragrance mixtures obtainable from vegetable sources, for example pine, citrus, jasmine, patchouli, rose or ylang-ylang oil. Also suitable are clary oil, camomile oil, clove oil, melissa oil, mint oil, cinnamon leaf oil, lime blossom oil, juniper berry oil, vetiver oil, olibanum oil, galbanum oil and labdanum oil and orange blossom oil, neroli oil, orange peel oil and  
10 sandalwood oil.

The detergent tablets according to the invention normally contain less than 0.01% by weight of dyes whereas perfumes can make up as much as 2% by weight of the formulation as a whole.

The perfumes may be directly incorporated in the detergents  
15 according to the invention, although it can also be of advantage to apply the perfumes to supports which strengthen the adherence of the perfume to the washing and which provide the textiles with a long-lasting fragrance through a slower release of the perfume. Suitable support materials are, for example, cyclodextrins, the cyclodextrin/perfume complexes optionally  
20 being coated with other auxiliaries.

In order to improve their aesthetic impression, the detergents according to the invention may be colored with suitable dyes. Preferred dyes, which are not difficult for the expert to choose, have high stability in storage, are not affected by the other ingredients of the detergents or by  
25 light and do not have any pronounced substantivity for textile fibers so as not to color them.

The tablets according to the invention are produced by first dry-mixing the ingredients - which may be completely or partly pregranulated - and then shaping/forming, more particularly tableting, the resulting mixture  
30 using conventional processes. To produce the tablets according to the

invention, the premix is compacted between two punches in a die to form a solid compactate. This process, which is referred to in short hereinafter as tableting, comprises four phases, namely metering, compacting (elastic deformation), plastic deformation and ejection.

5           The premix is first introduced into the die, the filling level and hence the weight and shape of the tablet formed being determined by the position of the lower punch and the shape of the die. Uniform dosing, even at high tablet throughputs, is preferably achieved by volumetric dosing of the premix. As the tableting process continues, the top punch comes into  
10       contact with the premix and continues descending towards the bottom punch. During this compaction phase, the particles of the premix are pressed closer together, the void volume in the filling between the punches continuously diminishing. The plastic deformation phase in which the particles coalesce and form the tablet begins from a certain position of the  
15       top punch (and hence from a certain pressure on the premix). Depending on the physical properties of the premix, its constituent particles are also partly crushed, the premix sintering at even higher pressures. As the tableting rate increases, i.e. at high throughputs, the elastic deformation phase becomes increasingly shorter so that the tablets formed can have  
20       more or less large voids. In the final step of the tableting process, the tablet is forced from the die by the bottom punch and carried away by following conveyors. At this stage, only the weight of the tablet is definitively established because the tablets can still change shape and size as a result of physical processes (re-elongation, crystallographic effects,  
25       cooling, etc.).

          The tableting process is carried out in commercially available tablet presses which, in principle, may be equipped with single or double punches. In the latter case, not only is the top punch used to build up pressure, the bottom punch also moves towards the top punch during the  
30       tableting process while the top punch presses downwards. For small



production volumes, it is preferred to use eccentric tablet presses in which the punch(es) is/are fixed to an eccentric disc which, in turn, is mounted on a shaft rotating at a certain speed. The movement of these punches is comparable with the operation of a conventional four-stroke engine.

5     Tabletting can be carried out with a top punch and a bottom punch, although several punches can also be fixed to a single eccentric disc, in which case the number of die bores is correspondingly increased. The throughputs of eccentric presses vary according to type from a few hundred to at most 3,000 tablets per hour.

10             For larger throughputs, rotary tablet presses are generally used. In rotary tablet presses, a relatively large number of dies is arranged in a circle on a so-called die table. The number of dies varies – according to model – between 6 and 55, although even larger dies are commercially available. Top and bottom punches are associated with each die on the  
15     die table, the tabletting pressures again being actively built up not only by the top punch or bottom punch, but also by both punches. The die table and the punches move about a common vertical axis, the punches being brought into the filling, compaction, plastic deformation and ejection positions by means of curved guide rails. At those places where the  
20     punches have to be raised or lowered to a particularly significant extent (filling, compaction, ejection), these curved guide rails are supported by additional push-down members, pull-down rails and ejection paths. The die is filled from a rigidly arranged feed unit, the so-called filling shoe, which is connected to a storage container for the premix. The pressure applied to  
25     the premix can be individually adjusted through the tools for the top and bottom punches, pressure being built up by the rolling of the punch shank heads past adjustable pressure rollers.

To increase throughput, rotary presses can also be equipped with two filling shoes so that only half a circle has to be negotiated to produce a  
30     tablet. To produce two-layer or multiple-layer tablets, several filling shoes

are arranged one behind the other without the lightly compacted first layer being ejected before further filling. Given suitable process control, jacketed and bull's-eye tablets – which have a structure resembling an onion skin – can also be produced in this way. In the case of bull's-eye tablets, the upper surface of the core or rather the core layers is not covered and thus remains visible. Rotary tablet presses can also be equipped with single or multiple punches so that, for example, an outer circle with 50 bores and an inner circle with 35 bores can be simultaneously used for tableting. Modern rotary tablet presses have throughputs of more than one million tablets per hour.

Tableting machines suitable for the purposes of the invention can be obtained, for example, from the following companies: Apparatebau Holzwarth GbR, Asperg, Wilhelm Fette GmbH, Schwarzenbek, Hofer GmbH, Weil, KILIAN, Cologne, KOMAGE, Kell am See, KORSCH Pressen GmbH, Berlin, Mapag Maschinenbau AG, Bern (Switzerland) and Courtoy N.V., Halle (BE/LU). One example of a particularly suitable tableting machine is the model HPF 630 hydraulic double-pressure press manufactured by LAEIS, D.

The tablets can be made in certain shapes and certain sizes. Suitable shapes are virtually any easy-to-handle shapes, for example slabs, bars, cubes, squares and corresponding shapes with flat sides and, in particular, cylindrical forms of circular or oval cross-section. This last embodiment encompasses shapes from tablets to compact cylinders with a height-to-diameter ratio of more than 1.

The portioned pressings may be formed as separate individual elements which correspond to a predetermined dose of the detergent/cleaner. However, it is also possible to form pressings which combine several such units in a single pressing, smaller portioned units being easy to break off in particular through the provision of predetermined weak spots. For the use of laundry detergents in machines of the standard

European type with horizontally arranged mechanics, it can be of advantage to produce the portioned pressings as cylindrical or square tablets, preferably with a diameter-to-height ratio of about 0.5:2 to 2:0.5. Commercially available hydraulic presses, eccentric presses and rotary  
5 presses are particularly suitable for the production of pressings such as these.

The three-dimensional form of another embodiment of the tablets is adapted in its dimensions to the dispensing compartment of commercially available domestic washing machines, so that the tablets can be  
10 introduced directly, i.e. without a dosing aid, into the dispensing compartment where they dissolve on contact with water. The cleaning tablets may of course also be used in conjunction with a dosing aid.

Another preferred multi-phase tablet which can be produced has a plate-like or slab-like structure with alternately thick long segments and thin  
15 short segments, so that individual segments can be broken off from this "bar" at the predetermined weak spots, which the short thin segments represent, and introduced into the machine. This "bar" principle can also be embodied in other geometric forms, for example vertical triangles which are only joined to one another at one of their longitudinal sides.

20 In another possible embodiment, however, the various components are not compressed to form a single tablet, instead the tablets obtained comprise several layers, i.e. at least two layers. These various layers may have different dissolving rates. This can provide the tablets with favorable performance properties. If, for example, the tablets contain components  
25 which adversely affect one another, one component may be integrated in the more quickly dissolving layer while the other component may be incorporated in a more slowly dissolving layer so that the first component can already have reacted off by the time the second component dissolves. The various layers of the tablets can be arranged in the form of a stack, in  
30 which case the inner layer(s) dissolve at the edges of the tablet before the

outer layers have completely dissolved. Alternatively, however, the inner layer(s) may also be completely surrounded by the layers lying further to the outside which prevents constituents of the inner layer(s) from dissolving prematurely.

5 In another preferred embodiment of the invention, a tablet consists of at least three layers, i.e. two outer layers and at least one inner layer, a peroxy bleaching agent being present in at least one of the inner layers whereas, in the case of the stack-like tablet, the two outer layers and, in the case of the envelope-like tablet, the outermost layers are free from peroxy  
10 bleaching agent. In another possible embodiment, peroxy bleaching agent and any bleach activators present and/or enzymes may be spatially separated from one another in one and the same tablet. Multilayer tablets such as these have the advantage that they can be used not only via a dispensing compartment or via a dosing unit which is added to the wash  
15 liquor, instead it is also possible in cases such as these to introduce the tablet into the machine in direct contact with the fabrics without any danger of spotting by bleaching agent or the like.

Similar effects can also be obtained by coating individual constituents of the detergent composition to be compressed or the tablet as  
20 a whole. To this end, the tablets to be coated may be sprayed, for example, with aqueous solutions or emulsions or a coating may be obtained by the process known as melt coating.

After pressing, the cleaning tablets have high stability. The fracture resistance of cylindrical tablets can be determined via the diametral fracture  
25 stress. This in turn can be determined in accordance with the following equation:

$$\sigma = \frac{2P}{\pi Dt}$$

30

where  $\sigma$  represents the diametral fracture stress (DFS) in Pa, P is the force

in N which leads to the pressure applied to the tablet that results in fracture thereof, D is the diameter of the tablet in meters and t is its height.

### Examples

5 Premixes were prepared by mixing surfactant-containing granules with powder-form additive components and were then tableted in a Korsch tablet press to form detergent tablets. The tableting pressure was adjusted to give two series of tablets differing in their hardness.

10 The surfactant granules A which led to the tablets E1 according to the invention contained 1.1% by weight APG and 6.4% by weight nonionic surfactant based on an ethoxylated alcohol (APG:ethoxylate ratio 1:5.82). The surfactant granules B which were tableted to form the tablets C1 contained only 0.5% by weight APG and 7.0% by weight ethoxylated alcohol (APG:ethoxylate ratio 1:14); the surfactant granules C which gave  
15 the tablets C2 contained exactly reversed quantities of APG and ethoxylated nonionic surfactant (APG:ethoxylate ratio 14:1).

Tablets E1, C1 and C2 and E1', C1' and C2' differ only in their hardness and not in their composition. The composition of the surfactant granules and the composition of the premixes to be tableted (and hence of  
20 the tablets) are shown in Tables 1 and 2.

**Table 1:** Composition of the surfactant granules [% by weight]

	<b>A</b>	<b>B</b>	<b>C</b>
C <sub>9-13</sub> alkyl benzenesulfonate	18.6	18.6	18.6
C <sub>12-18</sub> fatty alcohol + 7 EO	6.4	7.0	0.5
C <sub>12-18</sub> fatty alcohol sulfate	4.1	4.1	4.1
C <sub>12-16</sub> alkyl-1,4-glycoside	1.1	0.5	7.0
Soap	1.6	1.6	1.6
Optical brightener	0.8	0.8	0.8
Sodium carbonate	14.4	14.4	14.4
Sodium silicate	6.7	6.7	6.7
Acrylic acid/maleic acid copolymer	5.3	5.3	5.3
Zeolite A (water-free active substance)	27.6	27.6	27.6
Water, salts	Balance	Balance	Balance

**Table 2:** Composition of the premix [% by weight]

Surfactant granules	61.3
Zeolite	2.0
Sodium perborate monohydrate	23.7
TAED	2.4
Foam inhibitor	4.7
Polyacrylate	1.4
Perfume	0.5
Disintegration aid (cellulose*)	4.0

\* compacted cellulose (particle size: 90% by weight > 400 µm)

The hardness of the tablets was measured by deforming a tablet until it broke, the force being applied to the sides of the tablet and the maximum force withstood by the tablet being determined.

To determine tablet disintegration, a tablet was placed in a glass beaker filled with water (600 ml water, temperature 30°C) and the time taken for the tablet to disintegrate completely was measured.

The individual premixes were compressed to series of tablets in a rotary tablet press (manufacturer, model). The individual tablets of a series may vary in their hardness and disintegration times because, in rotary presses, the tableting pressure cannot be adjusted as exactly as it can in eccentric presses for example.

The experimental data are set out in Table 3:

**Table 3:**

Detergent tablets [physical data]

Tablet	E1	C1	C2	E1'	V1'	V2'
Tablet hardness	43-51N	38-45N	40-47N	51-59N	53-60N	52-61N
Tablet disintegration	15-22 secs.	30-37 secs.	40-50 secs.	23-27 secs.	42-48 secs.	>50 secs.